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Determination of Heavy Metals in Animal feed by Inductive Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

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ABSTRACT
A novel analytical method for accurate determination of heavy metal content in Animal Feed samples based on a relatively recent analytical technique, Inductive Couples Plasma- Optical emission spectrometry (ICP-OES), is developed in the present work. Microwave assisted acid digestion method is utilized to digest the animal feed samples. Precision and accuracy of the developed method are demonstrated using replicate analyses. The limit of quantification and detection of heavy metals are determined; the linear regression coefficient was greater than 0.998. Analytical wavelengths are selected according to the sensitivity and interference effects. The results obtained in this work demonstrate the potential of ICP-OES technique for the determination of heavy metal content in Animal Feed, which achieved lower detection limits, higher accuracy, and better reproducibility as compared to other techniques.

Keywords: *Animal Feed; microwave assisted acid digestion; Inductive coupled plasma optical emission spectrometry (ICP-PES); Heavy Metals*

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INTRODUCTION

Feed industry in India is about 50 years old. It primarily consists of cattle feed and poultry feed segments. Animal feed plays a vital role in the food chain as feed is one of the most crucial contributors in ensuring safe, abundant and affordable animal protein. Incessant population growth and rising affordability has surged demand for animal protein in India. Animal feed industry deals with food given to animals in cattle, poultry and aquaculture sector as part of animal husbandry. Indian feed industry predominantly caters to cattle and poultry feed segment. Although animal feed industry in India is almost five decades old, the industry is still in its infancy with high reliance on imports. However, the industry is very lucrative with large number of domestic players and various foreign multinationals striving to enter into the market.Growth of Indian animal feed market is propelled by rise in demand for animal protein, surge in dairy products consumption and growth of livestock population [1].

Commonly, animal manure was applied to agricultural land to improve the soil fertility and organic matter content. However, this practice also results in serious environmental problems, such as nitrate and phosphate contamination of surface waters [2]. Another important problem induced by animal manure application is metal pollution, as animal manure contains high metal (Cu, Zn, As, Cd) concentrations [3-5]. Residues of heavy metals in manures can be accumulated in surface soils as a result of long-term agricultural use [6-8].

Accumulation of heavy metals could not only affect the soil fertility and the product quality[9], but also promote metal migration through leaching and runoff [10-11]. Therefore, due to potential risks of heavy metal pollution heavy metal residues in animal manure have received scientific attention[12-13]. A wide range of heavy metals in animal manures has been investigated in intensive animal production.

Heavy metal toxicity is one of the major current environment health problems and is potentially dangerous because of bio-accumulation through the food chain [14] and this can cause hazardous effects on livestock and human health [15]. In general, the hazardous effects of these toxic elements depends upon the dietary concentration of the element, absorption of the element by the system, homeostatic control of the body for the element and also the species of the animal involved.

As Growth in the animal feed market, quality of animal feed is also important part. There is many methods to check physical and chemical properties of animal feed. In this research paper, we are going to find out metal content present in animal feeds. Common methods of chemical analysis of feeds for mineral micronutrients include atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) [11-13].

MATERIAL AND METHODS

Instrumentation

A ICP-OES (Model: Prodigy 7, Teledyne Leeman Labs.,USA) was used in all trace element determinations. The microwave-assisted acid digestion technique was utilized for the preparation of samples using the microwave assisted acid digestion system, CEM MARS 6. No further sample preparation was required and no modifiers or ionization buffers were added.

Reagents and Materials

All chemicals used were of analytical reagent quality. Suprapure HNO_3 (69%) from Merck (Darmstadt, Germany). Individual standard stock solutions of 1000 mg/L of Cu, Zn, Fe, P, Al, Ni, Co, Mn, Pb, Hg and As Merck (Darmstadt, Germany) were utilized. For calibrations, standards of appropriate dilutions were prepared using the individual stock standard solutions. Ultrapure water with a resistivity of 18.2 MW/cm was obtained using the Milli-Q system (Millipore, Billerica, MA, USA). Microwave assisted acid digestion system (CEM, Corp. MARS 6, Matthews, NC, USA) was utilized.

Sample

A total of 5 samples of five different types of manufacturers were purchased from different sources in the local markets of Mumbai.

1. Allen & Page Cattle and calf mix
2. Manna Pro Calf- Manna
3. TruCare 4 Top-dress Trace Mineral Blend for Livestock
4. Virbac Agrimin Forte Chelated
5. Gujarat Ambuja Exports ltd Cattle feed

2.4. Sample Preparation

Procedure- Sample Digestion- For microwave digestion, The CEM Mars 6 closed-vessel, microwave assisted acid digestion system (Matthews, NC, USA) was used to digest animal feed samples in acids. Samples were prepared by accurately weighing 0.5 g of each sample into a Teflon/PFA-lined microwave assisted acid digestion vessels and 8ml of an supraure Nitric acid was added in vessels. Digestion parameters are 190 degree temp, power 800 watts, hold time 25min with CEM Xpress vessels.

Metal Analysis-

At the end of the digestion program, the samples were quantitatively transferred to 50 mL volumetric flask and diluted with distilled water. The concentration of metals in the sample was determined using Inductive coupled plasma PRODIGY 7 instrument manufactured by Teledyne Leeman Lab. All quality control and assurance measures were taken including calibration check measures, determination of Method Quantification Limits (MQL), and replicate analysis of samples.

Table 1: LOD (limit of detection), LOQ(limit of quantification),linearity, selectivity, precision and assay these parameters covered in current studies.

Sr. No.	Metal name	LOD ppm	LOQ ppm	Linearity Correlation coefficient (r)	Precision Intraassay (%R.S.D.)	Precision Interday (%R.S.D.)
1	Sodium	0. 625ppm	1.5 ppm	0.9996	98.12%	97.34%
2	Calcium	0.625 ppm	1.5 ppm	0.9978	99.34%	98.54%
3	Phosphorus	0.625 ppm	1.5 ppm	0.9983	99.08%	98.43%
4	Zinc	0.625 ppm	1.5 ppm	0.9964	99.72%	98.66%
5	Copper	0.625 ppm	0.9 ppm	0.9991	99.77%	98.92%
6	Cobalt	0.3 ppm	0.9 ppm	0.9983	98.92%	98.07%
7	Magnesium	0.3 ppm	0.9 ppm	0.9964	99.72%	99.12%
8	Manganese	0.3 ppm	0.9 ppm	0.9991	99.52%	98.32%

Table 2: LOD, LOQ, Linearity, Intraassay and intraday precision, repeatability OF Inductive coupled plasma method for the determination of metals.

Sr. No.	Metal name	Sample Name				
		Allen & Page Cattle and calf mix	Manna Pro Calf- Manna	TruCare 4 Top-dress Trace Mineral Blend for Livestock	Virbac Agrimin Forte Chelated	Gujarat Ambuja Exports ltd Cattle feed
1	Sodium	4140 ppm	3560 ppm	2730ppm	1740 mg	2100ppm
2	Calcium	12560 ppm	10540 ppm	3500 ppm	25675ppm	5515ppm
3	Phosphorus	4670ppm	1300 ppm	1750 ppm	1150ppm	1050 ppm
4	Zinc	1340 ppm	355ppm	10893 ppm	9600 ppm	1640ppm
5	Copper	1750ppm	2720 ppm	10893 ppm	1200ppm	1320 ppm
6	Cobalt	1770 ppm	1430 ppm	6049 ppm	750 ppm	1210ppm
7	Magnesium	2300 ppm	3500 ppm	3750 ppm	6000 ppm	3280 ppm
8	Manganese	2780 ppm	3460 ppm	6000 ppm	3540 ppm	2650 ppm

Metal contents are in ppm.

CONCLUSION

The present method is applicable over a wider concentration range; where 0.5 -10 ppm solution of std. for Sodium, Calcium, Phosphorus, Zinc and 0.3 – 5 ppm solution of std. for Cobalt, Magnesium, Copper and Manganese can be determined, from animal feed. This is clear from the results obtained for the animal feed preparations that no interference of another matter. Sensitivity, accuracy, convenience and the reproducibility of the results are superior to those obtained from other methods like AAS, the method should be useful for routine analytical and quality control analysis.

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